N FR 2 552 308

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(12) UK Patent Application (19) GB (11) 2 148 117 A

(43) Application published 30 May 1985

(21) Application No 8424451

(22) Date of filing 27 Sep 1984

(30) Priority data

(31) 3450/83 (32) 28 Sep 1983 (33) AT
3434774 21 Sep 1984 DE

(51) INT CL[4]
A61K 9/46 A23L 2/40

(52) Domestic classification
A5B 828 831 832 836 L N
U1S 1317 2417 A5B

(56) Documents cited
None

(58) Field of search
A5B
A2B

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(54) **An effervescent composition suitable for effervescent tablets**

(57) An effervescent composition, more particularly for effervescent tablets, contains at least one solid crystalline organic acid and at least one carbonate which liberates CO_2 on reaction with the organic acid, the acid crystals having a coating which contains calcium carbonate and which adheres to their surface by means of a bonding layer formed by surface-reaction of the calcium carbonate containing coating material with a surface layer of each acid crystal. The effervescent tablets may also contain minerals and/or vitamins or acetylsalicylic acid, the latter being present, optionally, with paracetamol in a two-layer structure.

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SPECIFICATION

An Effervescent Composition, Suitable for Effervescent Tablets, Effervescent Tablets Made from such a Composition, and a Process for the Preparation Thereof

This invention relates to an effervescent composition suitable for preparing effervescent tablets, and to an effervescent tablet made using such a composition, and a process for its preparation.

It is known to prepare effervescent tablets from a system based on the reaction of an organic acid, e.g. citric acid or tartaric acid, with a CO_2 liberating substance, such as sodium bicarbonate, sodium carbonate, potassium bicarbonate or potassium carbonate. A common criticism of such systems is that an extremely high proportion of sodium ions are present in the resultant composition and it is desirable to devise an effervescent system containing fewer or the fewest possible number of sodium ions. Whilst it is possible to use potassium carbonate or bicarbonate instead of sodium, one obstacle to using potassium bicarbonate and potassium carbonate alone is that this results in the resultant composition having an unpleasant soapy aftertaste. Additionally, the moisture-sensitivity resulting from the use of potassium salts causes considerable technical problems.

Whilst it would be desirable to find other CO_2-liberating bases, for example, calcium carbonate and magnesium carbonate, calcium carbonate, has heretofore been difficult to use, if used at all, because it reacts very slowly with organic acids and would therefore result in an effervescent system that requires far too long to dissolve.

An object of this invention is to provide an effervescent composition, a process for the preparation of such a composition and an effervescent tablets capable of preparation using said composition, in which the effervescent reaction is so accelerated that the dissolution times are not excessively long, while the effervescent tablets which can be made are stable and capable of storage.

Accordingly, the invention provides an effervescent composition suitable for use in the preparation of effervescent tablets, which composition comprises at least one crystalline organic acid and at least one carbonate which liberates CO_2 on reaction with the organic acid, the acid crystals having a surface coating which contains calcium carbonate and which adheres to their surface by means of a bonding layer formed by surface-reaction of the calcium carbonate containing coating material with a surface layer of each acid crystal.

Preferably, the bonding layer covers at least 80%, more particularly at least 95%, of the surface of the acid crystals. This complete covering and the bonding of the calcium carbonate to the surface results in the unexpectedly fast reaction between the acid, e.g. citric acid, and calcium carbonate, a reaction which would otherwise take place much more slowly. The extensive covering of the acid crystals also gives reliable passivation thereof in respect of ambient moisture. One particular effect resulting from this feature is that no external bonding means have to be used whatsoever for the system structure, i.e., the adhesion of the components is obtained solely by means of the bonding layer.

Conveniently, the particle size of the calcium carbonate is approximately one order of magnitude smaller than that of the acid crystals to give the most coherent coating possible on the acid crystals.

Preferably, the bonding layer consists of 5% by weight maximum, more particularly 2% by weight maximum, of the calcium carbonate in the form of the acid salt, so that the maximum amount of calcium carbonate is available for the effervescent reaction.

The calcium carbonate component of the effervescent mixture preferably has a particle size of 20μ maximum, because the reaction surface of the calcium carbonate is consequently very large and the resulting reaction speed is comparable to sodium bicarbonate.

Although the coating material preferably consists solely of calcium carbonate, it may also contain sodium and/or potassium hydrogen carbonate. The surface coating may be in the form of a plurality of layers and, in one embodiment, one such layer contains basically potassium and/or sodium salts and another layer basically calcium salts.

The surface coating may comprise a surface-reacted calcium carbonate layer connected to the surface of the acid crystals by way of the bonding layer, and a potassium hydrogen carbonate layer adhering to the calcium carbonate layer.

Since the bonding layer in an effervescent composition of the invention replaces the bonding agent conventionally used in powder technology, it is also possible to use this bonding layer for the incorporation of mineral substances and/or vitamins.

In a further aspect the invention provides an effervescent tablet prepared using an effervescent composition of the invention.

Such an effervescent tablet may also contain vital mineral substances and vitamins. The invention also provides for the use of such effervescent tablets for the mineralization of soft drinks.

The use of effervescent tablets embodying the invention for the mineralization of soft drinks is very advantageous because soft drinks, which have a very high sugar content and are in some cases extremely deficient in mineral salts, in conjunction with the excessive use of common salt [sodium chloride] means that the organism is increasingly deprived of vital minerals. The effervescent tablets according to the invention thus enable large quantities of calcium, magnesium, potassium and other vital ions to be conveniently incorporated in a soft drink in conjunction with a very low sodium ion content, if any.

Such an effervescent tablet may contain at least one inert extender, such as mannitol or the like, and, desirably, the ratio of effervescent composition to extender, by weight, is about 1:1.

One preferred effervescent tablet may comprise an effervescent composition, wherein the said coating is free of sodium ions and consists of a first calcium carbonate containing layer, a second potassium hydrogen carbonate containing layer, and a third fumaric acid containing layer, and of acetyl salicylic acid.

The effervescent tablet may have a structure consisting of at least two layers of different compositions.

If required, just one of the tablet layers may contain an effervescent composition of the invention. In this case the tablet layer which does not contain an effervescent composition may contain at least one active substance.

Desirably, one of the tablet layers may contain paracetamol and one of the tablet layers may contain acetylsalicylic acid.

In a further aspect, the invention provides a process for the preparation of an effervescent composition which process comprises the steps of intermixing the or each organic acid and the or each carbonate in a vacuum mixing machine with a mixture of ethanol and water, some of the or each organic acid and some of the mixture of ethanol and water first being mixed at a temperature of about 60°C and a pressure of about 0.1 bar or less; and subsequently introducing calcium carbonate such that a surface-reaction between the calcium carbonate and the acid crystals occurs in at least one first surface-reaction step until the pressure in the vacuum mixing machine has risen to about 0.9 bar as a result of the CO_2 gas evolved during the surface-reaction.

Desirably, when the calcium carbonate coating of the acid crystals is complete, another surface-reaction takes place with the addition of the residue of the or each organic acid and of potassium bicarbonate and the remainder of the mixture of alcohol and water, under the temperature and pressure conditions of the first surface-reaction step.

In an embodiment particularly suitable for the production of an effervescent mixture suitable for the preparation of salicylic acid containing effervescent tablets, when the calcium carbonate coating of the acid crystals is complete a second surface-reaction is carried out with the addition of potassium hydrogen carbonate and the remaining ingredients, whereupon after drying at about 0.9 bar the resulting granulate particles are coated with fumaric acid in a micronized form.

The invention is based upon the surprising finding that the effervescent reaction between an organic acid, such as citric acid, and calcium carbonate can be accelerated by coating crystals of the organic acid with calcium carbonate and thus creating a very intimate contact between the calcium carbonate and the acid at the surface of the crystals. The resulting reaction speed achieved between the acid and calcium carbonate is comparable to the reaction speed previously obtainable with carbonates or bicarbonates of the alkali metals.

To achieve the desired coating of the organic acid crystals, it is necessary to use a binder which, conveniently, constitutes a product of a surface-reaction of about 5—10% of the calcium carbonate at the surface of the acid, e.g. citric acid, with the resultant formation of a calcium salt corresponding to the acid. The surface-reaction between calcium carbonate and acid, e.g., citric acid, serves to anchor the remaining calcium carbonate rigidly to the surface of the acid crystal so that this structure does not separate even during a subsequent mixing operation.

The bonding mechanism is preferably obtained by pre-wetting acid crystals of different sizes, e.g. from 50µm to 500µm, with a mixture of alcohol and water, then evacuating the wetted crystals to a pressure of 500 mbar, e.g. in a vacuum mixer, sucking in the calcium carbonate, and starting mixing at 500 mbar vacuum, preferably in an agitator which rocks about its horizontal axis and which in each case agitates against gravity. Such three-dimensional mixing movement causes the pre-wetted acid crystals to be brought into contact with the calcium carbonate. It is possible to measure the resulting reaction by measuring the drop of the vacuum present in the vacuum mixer using a suitable meter. After a certain amount of gas has been evolved the reaction is stopped by applying full vacuum, and the resulting monocalcium citrate layer serves as a binder for the calcium carbonate attached to the surface of the acid crystals by moisture. Subsequent removal of the moisture causes the system to remain mechanically and chemically stable.

Using this method it is possible to introduce quantities of calcium carbonate corresponding approximately to the stoichiometric amount of 1 mole of citric acid to 1 mole of calcium carbonate, only 5—10% of the calcium carbonate being reacted to produce the binder.

Manufacture in vacuo allows, for the first time, accurate control of the progress of the reaction and enables the reaction to be exactly terminated at any time with accurate reproducibility of the process. Moreover, a correspondingly slow rate of agitation results in an unobstructed build-up of the system and not its destruction as would be the case in other processes, e.g. fluidized bed drying. In particular, manufacture in vacuo ensures—as already stated—that an effervescent tablet prepared from the composition that has a low or zero sodium content dissolves rapidly like a conventional tablet based on sodium hydrogen carbonate and, in addition, is much less sensitive to moisture.

Whilst this provides a usable effervescent system, it is also possible to add desired quantities of potassium bicarbonate or potassium carbonate, and/or a small quantity of sodium carbonate, the amount added depending upon whether the system is to be designated as "low sodium" or "very low sodium".

When selecting the organic acid, those acids which yield insoluble calcium salts, e.g. tartaric acid, should not be selected. On the other hand, it is possible to use malic acid, fumaric acid, adipic acid, and any other suitable acid.

Obviously, an effervescent tablet embodying the invention may contain other conventional additives, including extenders such as mannitol or the like.

Particularly suitable protocols and apparatus for the production of effervescent systems embodying the invention are described in Austrian Patent No. 376147.

Further features and advantages of the invention will be apparent from the following description, in which exemplified embodiments are explained in detail.

EXAMPLE 1

22 parts of citric acid of a particle size between 0.4 and 0.6 mm, and 43 parts of citric acid of particle size of 0.1 mm. were mixed together, heated to 40°C, and 10 parts of 50% ethanol were added. After vibratory mixing for 5 minutes, evacuation was carried out to 500 mbar, using a vacuum pump connected with the mixer *via* a valve, and 20 parts of micronized calcium carbonate were introduced. Evacuation was repeated without agitation, and on reaching 500 mbar mixing was carried out with vibration and with the valve to the vacuum pump shut off. The resulting reaction caused the vacuum to drop slowly and the reaction was allowed to continue until the vacuum dropped to 200 mbar at which time a full vacuum was applied. The difference in the supernatant volume, allowing for the pressure difference, gave a reaction of about 4% of the corresponding amount of calcium carbonate to monocalcium citrate.

Agitation was suspended on reaching 800 mbar and drying was carried out to a pressure of 10 mbar with intermittent agitation.

Up to 80 parts of potassium bicarbonate and up to 30 parts of sodium carbonate can also be mixed in this system if desired. However, the system enables preparation of effervescent tablets which are usable on their own.

A composition prepared as above provides a base that is particularly suitable for the preparation of high-dosage acetylsalicylic acid effervescent tablets which, if taken repeatedly, contain a well-balanced amount of alkali-metal and alkaline-earth ions, whereas a very large quantity of sodium ions was hitherto necessary to obtain the desired effervescent effect.

EXAMPLE 2

22 parts of citric acid of a size of 0.5 mm were mixed with 88 parts of citric acid of particle size of 0.1 mm and the mixed particles were wetted with 20 parts of ethanol/water. 22 parts of calcium carbonate were introduced and after drying 60 parts of potassium bicarbonate and 10 parts of sodium carbonate were added.

Depending upon the amount of acetyl salicylic acid t be added, it is possible also to introduce up to 40 parts of lactose, the corresponding effervescent system being diluted in order to increase the stability of the acetylsalicylic acid. It is possible to prepare 4g effervescent tablets using this composition, which may contain up to 1g of acetylsalicylic acid as a single dose.

EXAMPLE 3

40 parts of citric acid of particle size 0.7 mm were mixed with 30 parts of ascorbic acid and another 45 parts of pulverized citric acid were added.

25 parts of a mixture of 70% ethanol and 30% water was used for wetting the acid crystals and the reaction was carried out with 25 parts of calcium carbonate as in Example 1.

After drying to 10 mbar, 80 parts of potassium bicarbonate and 60 parts of lactose with a particle size of 0.2 mm were added and the resultant composition was compressed into tablets.

Thus formed low-sodium vitamin-C effervescent tablets have substantially the same speed of dissolution as those previously made with sodium bicarbonate.

EXAMPLE 4

22 parts by weight of citric acid of a particle size between 0.4 mm and 0.6 mm and 30 parts by weight of citric acid of particle size 0.1 mm were mixed, heated to 40°C and a solution of 13 parts by weight of gluconic acid deltalactone in 5 parts by weight of water was added. The reaction was carried out and the composition dried in the same way as in Example 1.

The advantage of adding gluconic acid deltalactone, in which 1 part by weight of the lactone converts to gluconic acid, is that the speed of dissolution of the resultant composition is accelerated, since the different pH values of citric acid and gluconic acid prevent any surface buffering which would retard the reaction.

EXAMPLE 5

200 parts of crystallized citric acid were wetted with 5 parts of ethanol and 5 parts of water and heated to 60°C in a vacuum mixer. 30 parts of calcium carbonate were then added to the mixer and the mixture was allowed to react, evacuation initially being to about 100 mbar and the empty volume of the vacuum mixer being allowed to fill with CO_2 gas developing from the reaction to a pressure of 900 mbar.

The gas evolution was repeated for a second time and then stopped by applying a full vacuum.

Subsequently, 10 parts of potassium bicarbonate were added and another 20 parts of citric acid, passivation being repeated with 2 parts of ethanol and 1 part of water.

The result was a passivated effervescent composition consisting of surface-reacted calcium carbonate and potassium bicarbonate, with an appreciable stability in respect of moisture.

The amounts of sodium bicarbonate statutorily allowed whilst retaining a "low sodium" designation can also be added to this basic effervescent composition. Together with a conventional multivitamin mix, the composition gives a readily compressible effervescent tablet with an extremely low sodium ion content.

EXAMPLE 6

105 parts of ascorbic acid [vitamin C], and 130 parts of citric acid were heated to 60°C together with

6 parts of ethanol and 3 parts of water, and treated with 22 parts of calcium carbonate as in Example 1. In this case, the ascorbic acid is also reacted superficially with calcium carbonate, thus greatly increasing the stability of the system, since the ascorbic acid with its low pH and easy water-solubility was already passivated at the surface and thus stabilized.

Again 10 parts of potassium bicarbonate and 10 parts of citric acid were reacted, so that all the exposed surface areas of the acids were passivated with calcium or potassium salts by surface-reaction. Here again drying was carried out towards the end at a pressure of at least 20 mbar and flavouring and colouring substances can be added dry in known manner and then compression carried out to form a tablet.

EXAMPLE 7

This example illustrates the more difficult preparation of low sodium salicylic acid-containing effervescent tablets:

68 parts of citric acid were wetted with 2 parts of ethanol and 1 part of water, heated to 60°C and reacted with 20 parts of calcium carbonate. Immediately thereafter, 40 parts of potassium hydrogen carbonate were reacted, but only once, 2 parts of 70% ethanol preferably being introduced to start the reaction.

After partial drying 20 parts of fumaric acid were added in micronized form in order to coat any still exposed surfaces of the reactants with fumaric acid. A particularly elegant procedure for this is for the preceding reactants to be dried only to a certain vacuum value, e.g. 90 mbar, thus leaving a slight residual moisture which keeps the micronized fumaric acid fixed at the surface.

This mix can be mixed with salicylic acid in a ratio of 2:1 and compressed to give hard rapidly disintegrating, effervescent tablets which contain no sodium ions at all and which are distinguished by low saponification of the free salicylic acid, even when stored for relatively long periods.

To improve the rate of disintegration, small quantities of sodium bicarbonate can of course be added in accordance with the relevant regulations. It is also possible to supplement or dilute the effervescent composition with inert substances, e.g. mannitol, this giving quicker disintegration times and better stability. A 1:1 ratio of mannitol to effervescent composition still gives quickly disintegrating and stable effervescent systems.

EXAMPLE 8

With a process of the invention it is also possible to manufacture even more complex effervescent tablets, this being advantageous particularly in the case of a tablet comprising normally incompatible substances. For example, the incompatible system comprising paracetamol and acetylsalicylic acid can be combined as indicated below in a two-layer effervescent tablet.

68 parts of citric acid mixed with 2 parts of ethanol, wetted with 1 part of water and heated to 60°C, were reacted with 20 parts of calcium carbonate as in Example 6.

Before the reaction was stopped, 20 parts of paracetamol were introduced, and immediately became lodged on the surface because of the tacky bonding force of the resulting calcium nitrate. Only then was the product dried in vacuo and the reaction then completed by allowing 40 parts of potassium hydrogen carbonate to react once with two parts of 70% ethanol. Here again, after partial drying to only 100 mbar, for example, 20 parts of fumaric acid were added in micronized form.

The resulting base for a paracetamol effervescent tablet in the form of a two-layer tablet can be compressed with a final mixture in the proportions indicated in Example 7 to form an acetylsalicylic acid containing effervescent tablet, only approximately 0.8% of the acetylsalicylic acid being lost in the form of free salicylic acid in the two-layer tablet, going to the compressed interface between the two phases of the two-layer tablet.

Because of the extremely good effervescent properties of the paracetamol base, however, the procedure may alternatively be used to produce a two-layer tablet in which, for example, the basic mixture is such as to give a 2.8g effervescent tablet containing the corresponding amount of paracetamol, whereupon a second layer, of an acetylsalicylic acid mixture consisting of 200 mg of acetylsalicylic acid and 500 mg of ordinary lactose, is pressed thereon to give a two-layer tablet having a total weight of 3.5g. Although the acid in this case is in a non-effervescent form, the effervescent action of the paracetamol-containing layer is sufficient to give complete dissolution of the acetylsalicylic acid in the total tablet. The extraordinary advantages of this system are that the paracetamol is fully stable in the low sodium content effervescent phase but prevents saponification effects of both the paracetamol and the Na-free effervescent free mixture on the aspirin. In this way it is possible to manufacture effervescent tablets with incompatible constituents, by means of a simple two-layer tablet press, to have a low sodium content.

EXAMPLE 9

With the procedure described it is also possible to make sodium-free or low sodium content effervescent tablets enriched in minerals and vitamins, e.g. of the B group:

500 parts of citric acid, preferably of a particle size of 0.2—0.3 mm, were heated with 30 parts of magnesium oxide and 150 parts of calcium carbonate to 60°C.

A solution of 40 parts of citric acid in 20 parts of water was added and the mixture was left to react until the supernatant evacuated space, the volume of which generally corresponds to twice the amount of citric acid, had been filled with CO_2.

A high vacuum was then applied and drying was carried out to a value of 100 mbar. 20 parts of iron sulphate, 40 parts of potassium citrate, 10 parts of potassium chloride and a corresponding quantity of the vitamin group B1, B2, BP and B6 were added to the still residually moist mixture.

The residually moist but passivated basic mixing results in the additives being bonded together so that after evacuation of the material to 10—20 mbar a uniform product is obtained which is easily pourable and, in particular, very stable to atmospheric moisture.

This product can either be used as a granulate in sachets for the production of instant sports drinks or can be compressed with an addition of 25% micronized fumaric acid to form effervescent tablets.

The features of the invention disclosed in the above description, in the drawing and in the claims, may, both individually and in any combination whatsoever, be significant to embodiment of the invention in its various aspects.

CLAIMS

1. An effervescent composition suitable for use in the preparation of effervescent tablets, which composition comprises at least one crystalline organic acid and at least one carbonate which liberates CO_2 on reaction with the organic acid, the acid crystals having a surface coating which contains calcium carbonate and which adheres to their surface by means of a bonding layer formed by surface-reaction of the calcium carbonate containing coating material with a surface layer of each acid crystal.

2. An effervescent composition according to Claim 1, wherein the bonding layer covers at least 80% of the surface of the acid crystals.

3. An effervescent composition according to Claim 2, wherein the bonding layer covers at least 95% of the surface of the acid crystals.

4. An effervescent composition according to Claim 1, 2 or 3, wherein the particle size of the calcium carbonate is approximately one order of magnitude smaller than that of the acid crystals.

5. An effervescent composition according to any one of Claims 1 to 4, wherein the bonding layer contains a maximum of 5% by weight, preferably a maximum of 2% by weight, of calcium carbonate in the form of the acid salt.

6. An effervescent composition according to any one of Claims 1 to 5 wherein the coating is made up of a number of layers.

7. An effervescent composition according to any one of the preceding claims, wherein the coating material also contains sodium and/or potassium hydrogen carbonate.

8. An effervescent composition according to claim 6 or 7, wherein one layer contains basically potassium and/or sodium salts and an outer layer contains basically calcium salts.

9. An effervescent composition according to any one of Claims 1 to 8, wherein the coating comprises a surface-reacted calcium carbonate layer connected to the surface of the acid crystals via the bonding layer, and a potassium carbonate layer adhering to the calcium carbonate layer.

10. An effervescent composition according to any one of the preceding claims, wherein the calcium carbonate has a maximum particle size of 20µ.

11. An effervescent tablet made from an effervescent mixture according to any one of Claims 1 to 10.

12. An effervescent tablet according to Claim 11, further comprising vital mineral substances and/or vitamins.

13. An effervescent tablet according to Claim 12, comprising an effervescent composition, wherein the said coating is free of sodium ions and comprises a first calcium carbonate containing layer, a second potassium hydrogen carbonate containing layer, and a third fumaric acid containing layer, and also contains acetylsalicylic acid.

14. An effervescent tablet according to any one of Claims 10 to 13, having a structure consisting of at least two layers of different compositions.

15. An effervescent tablet according to Claim 14, wherein only one of the tablet layers contains an effervescent composition of any one of Claims 1 to 9.

16. An effervescent tablet according to Claim 14, wherein the tablet layer which contains no effervescent composition contains at least one active substance.

17. An effervescent tablet according to Claim 14, 15 or 16, wherein one of the tablet layers contains paracetamol and one of the tablet layers contains acetylsalicylic acid.

18. A process for the preparation of an effervescent composition according to any one of Claims 1 to 10, which process comprises the steps of intermixing the or each organic acid and the or each carbonate in a vacuum mixing machine with a mixture of ethanol and water, some of the or each organic acid and some of the mixture of ethanol and water first being mixed at a temperature of about 60°C and a pressure of about 0.1 bar or less; and subsequently introducing calcium carbonate such that a surface-reaction between the calcium carbonate and the acid crystals occurs in at least one first surface-reaction step until the pressure in the vacuum mixing machine has risen to about 0.9 bar as a result of the CO_2 gas evolved during the surface-reaction.

19. A process according to Claim 18, wherein, when the calcium carbonate coating of the acid crystals is complete, another surface-reaction takes place with the addition of the residue of the or each organic acid and of potassium bicarbonate and the remainder of the mixture of alcohol and water, under the temperature and pressure conditions of the first surface-reaction step.

20. A process according to Claim 18, suitable for the preparation of an effervescent mixture suitable for the production of the effervescent tablet of Claim 13, wherein when the calcium carbonate coating of the acid crystals is complete a second surface-reaction is carried out with the addition of potassium hydrogen carbonate and the remaining ingredients, whereupon after drying at about 0.9 bar the resulting granulate particles are coated with fumaric acid in micronized form.

21. A soft drink comprising an effervescent composition according to any one of Claims 1 to 9.

22. A soft drink comprising an effervescent tablet according to any one of Claims 10 to 17.

23. Any novel feature or combination of features described herein.

Printed in the United Kingdom for Her Majesty's Stationery Office, Demand No. 8818935, 5/1985. Contractor's Code No. 6378.
Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.